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IS 500 (1999): Potassium metabisulphite, photographic grade
[CHD 5: Electroplating Chemicals and Photographic Materials]



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IS 500 : 1999

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(चौथा पुनरीक्षण)

Indian Standard

POTASSIUM METABISULPHITE, PHOTOGRAPHIC
GRADE — SPECIFICATION
(*Fourth Revision*)

ICS 37.040.30

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

March 1999

Price Group 4

FOREWORD

This Indian Standard (Fourth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Electroplating Chemicals and Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

This Indian Standard was first published in 1953 and subsequently revised in 1963 and 1972. The third revision of this standard was brought out in 1980 incorporating requirements for clarity of aqueous solution, pH, reaction to ammoniacal silver nitrate solution and particle size. The requirement for potassium metabisulphite was raised to 95 percent.

In this revision, the requirements for description and thiosulphate content have been modified. The method for determination of thiosulphate has also been suitably modified. The requirement for particle size has been made an alternate requirement.

Composition of the Committee responsible for the formulation of this standard is given in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard***POTASSIUM METABISULPHITE, PHOTOGRAPHIC
GRADE — SPECIFICATION***(Fourth Revision)***1 SCOPE**

This standard prescribes the requirements and methods of sampling and test for potassium metabi-sulphite, photographic grade.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards:

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid (<i>second revision</i>)
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
1266 : 1993	Sulphuric acid (<i>third revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
4905 : 1968	Methods for random sampling

3 REQUIREMENTS**3.1 Description**

The material shall be in the form of white, glassy crystals, and shall essentially consist of potassium metabisulphite ($K_2S_2O_5$) having relative molar mass 222.3.

3.2 The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of Table 1.

Table 1 Requirements for Potassium Metabisulphite, Photographic Grade
(*Clauses 3.2, A-3.3.1, A-4.3.1 and A-5.3.1*)

SI No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Annex A)
(1)	(2)	(3)	(4)
i)	Potassium metabisulphite, percent by mass. <i>Min</i>	95.0	A-1
ii)	Matter insoluble in water, percent by mass. <i>Max</i>	0.5	A-2
iii)	Iron (as Fe), percent by Mass. <i>Max</i>	0.005	A-3
iv)	Heavy metals (as Pb), percent by mass. <i>Max</i>	0.005	A-4
v)	Thiosulphate (as $K_2S_2O_4$), percent by mass. <i>Max</i>	0.07	A-5
vi)	Clarity of 20 percent aqueous solution	Clear and colourless, free from any suspended matter	A-6
vii)	pH of 5 percent aqueous solution	4.0 to 4.5	A-7
viii)	Reaction to ammoniacal silver nitrate	To pass test	A-8

3.3 Alternate Requirement

The material shall be of the following particle sizes when tested according to the method prescribed in Annex B:

	<i>Particle Size</i>	<i>Requirement</i>
a)	Retained on 850-micron IS Sieve, percent by mass. <i>Max</i>	5
b)	Passing through 850-micron IS Sieve and retained on	85

<i>Particle Size</i>	<i>Requirement</i>
150-micron IS Sieve, percent by mass, <i>Min</i>	
c) Passing through 150-micron IS Sieve, percent by mass, <i>Max</i>	10

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable containers with tight closures, preferably in glass bottles, as agreed to between the purchaser and the supplier. It shall be kept in a cool and dry place.

4.2 Marking

The containers shall be securely closed and marked with the following information:

- Name and description of the material,
- Indication of the source of manufacture,
- Mass of the material in the container,
- Year of manufacture, and
- Batch number in code or otherwise to enable the batch of manufacture to be traced from records.

4.2.1 BIS Certification Marking

The product may also be marked with the Standard Mark.

4.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in Annex C.

5.2 Criteria for Conformity

A lot shall be declared as conforming if the characteristics tested on the test sample as detailed in **C-4.1** satisfy the requirements given in this specification.

6 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (see IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(Clause 3.2)

METHODS OF TEST FOR POTASSIUM METABISULPHITE, PHOTOGRAPHIC GRADE

A-1 DETERMINATION OF POTASSIUM METABISULPHITE

A-1.1 Reagents

A-1.1.1 *Standard Iodine Solution* — 0.1 N, freshly standardized.

A-1.1.2 *Concentrated Hydrochloric Acid* — See IS 265.

A-1.1.3 *Standard Sodium Thiosulphate Solution* — 0.1 N, freshly standardized.

A-1.1.4 Starch Solution

Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water. Pour the resulting paste into one litre of boiling water, boil for 3 min, allow the solution to cool and decant off the clear supernatant liquid.

A-1.2 Procedure

Weigh accurately about 0.25 g of the material and add it to exactly 50 ml of standard iodine solution. Allow to stand for 5 min, add 1 ml of concentrated hydrochloric acid and titrate against standard sodium thiosulphate solution, adding starch indicator solution towards the end of the titration.

A-1.3 Calculation

Potassium metabisulphite ($K_2S_2O_3$), percent by mass
$$= \frac{5.56 (50 N_1 - V N_2)}{M}$$
 where

- N_1 = normality of standard iodine solution,
 V = volume in ml of standard sodium thiosulphate solution,
 N_2 = normality of standard sodium thiosulphate solution, and
 M = mass in g of the material taken for the test

A-2 DETERMINATION OF MATTER INSOLUBLE IN WATER**A-2.1 Reagents**

A-2.1.1 Ammonium Oxalate Solution — 4 percent (*m/v*).

A-2.1.2 Diammonium Hydrogen Phosphate Solution — 10 percent (*m/v*).

A-2.1.3 Dilute Ammonium Hydroxide — 10 percent (*m/m*) and 2.5 percent (*m/m*).

A-2.2 Procedure

Dissolve 10 g of the sample in 75 ml of water. Add 10 ml ammonium oxalate solution, 4 ml of diammonium hydrogen phosphate solution and 20 ml of 10 percent ammonium hydroxide. Allow to stand at least for 8 h. If any precipitate is formed, filter and wash with 2.5 percent ammonium hydroxide. Dry and ignite at about 800°C /h. Cool in a desiccator and weigh.

A-2.3 Calculation

Insoluble matter (and calcium, magnesium and ammonium precipitate), percent by mass

$$= \frac{100 m}{M}$$

where

m = mass in g of the residue, and

M = mass in g of the material taken for the test.

A-3 TEST FOR IRON**A-3.1 Apparatus**

A-3.1.1 Nessler Cylinder — 50-ml capacity.

A-3.2 Reagents

A-3.2.1 Concentrated Hydrochloric Acid — See IS 265.

A-3.2.2 Ammonium Persulphate

A-3.2.3 Butanolic Potassium Thiocyanate Solution

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-3.2.4 Dilute Sulphuric Acid — approximately 10 percent.

A-3.2.5 Standard Iron Solution

Dissolve 0.702 g of ferrous ammonium sulphate $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ in 10 ml of dilute sulphuric acid, and dilute with water to 1 000 ml. Take 10 ml of this solution and dilute to 100 ml. One millilitre of the final solution contains 0.01 mg of iron (as Fe).

A-3.3 Procedure

Dissolve 1.00 g of the material in 30 ml of hot water, add 5 ml of concentrated hydrochloric acid and evaporate to dryness on a water-bath. Add 15 ml of hot water and 2 ml of concentrated hydrochloric acid and evaporate again to complete dryness. Dissolve the residue in 10 ml of water and transfer completely to a Nessler cylinder, washing the dish with the minimum quantity of water. Add 1 ml of concentrated hydrochloric acid, about 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake vigorously for 30 s and allow to separate. Carry out a control test in another Nessler cylinder, using 5 ml of standard iron solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour after 5 min.

A-3.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the red colour produced in the butanolic layer with the material is not greater than that produced in the control test.

A-4 TEST FOR HEAVY METALS**A-4.1 Apparatus**

A-4.1.1 Nessler Cylinder — 50-ml capacity.

A-4.2 Reagents

A-4.2.1 Concentrated Hydrochloric Acid — See IS 265.

A-4.2.2 Concentrated Nitric Acid — See IS 264.

A-4.2.3 Standard Lead Solution

Weigh 1.600 g of lead nitrate and add to it 50 ml of concentrated nitric acid. Dissolve in water and make up the solution to 1 000 ml mark. Pipette out 10 ml of the solution and dilute it again with water to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of lead (as Pb). The diluted solution should be freshly prepared.

A-4.2.4 Acetic Acid — Approximately 1 N, lead-free.

A-4.2.5 Hydrogen Sulphide Solution — Saturated and freshly prepared.

A-4.3 Procedure

Dissolve 5.00 g of the material in 20 ml of hot water. Add 6 ml of concentrated hydrochloric acid and evaporate the contents nearly to dryness on a water-bath. Add 15 ml of hot water and 3 ml of concentrated hydrochloric acid. Again evaporate on the steam-bath, and then heat for 1 h at 150°C. Dissolve the residue in water, filter and dilute the filtrate to 50 ml. Take 10 ml of the solution in a Nessler cylinder and add 1 ml of acetic acid. Carry out a control test in another Nessler cylinder using 5 ml of standard lead solution in place of the solution of the material. Add 10 ml of hydrogen sulphide solution to both the cylinders, dilute with water to 50-ml mark and compare the colour produced in the two cylinders.

A-4.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

A-5 TEST FOR THIOSULPHATE

A-5.1 Reagents

A-5.1.1 Acetic Acid Solution — Approximately 2 M.

A-5.1.2 Formaldehyde — Approximately 360 g/l solution and neutralized to phenolphthalein.

A-5.1.3 Iodine — 0.05 M standard volumetric solution, 12.7 g of iodine per litre.

A-5.1.4 Sodium Thiosulphate — 0.1 M standard volumetric solution.

A-5.1.5 Sulphuric Acid — 0.05 M standard volumetric solution.

A-5.1.6 Starch Indicator Solution

Stir 5 g of soluble starch with 100 ml of a 10 g/l salicylic acid solution. Then add 300 to 400 ml of boiling water, boil until the starch dissolves then finally dilute to 1 000 ml with water.

A-5.1.7 Phenolphthalein Indicator Solution — 5 g/l.

Dissolve 5 g of phenolphthalein in 500 ml of ethanol and add 500 ml of water, with constant stirring. Filter if necessary.

A-5.1.8 Thiosulphate Standard Solution

Dilute 5 ml of the sodium thiosulphate solution (see A-6.1.4) to 1 000 ml.

A-5.1.9 Mercury (II) Chloride Solution

Dissolve 25 g of potassium bromide and 25 g of mercury (II) chloride in 900 ml of water at 50 °C. Cool, dilute to 1 000 ml and allow to stand overnight. Filter if not perfectly clear.

A-5.2 Apparatus

A-5.2.1 Graduated Pipette — 1-ml capacity.

A-5.2.2 Nessler Cylinders — 50-ml capacity.

A-5.3 Procedure

Weigh, to the nearest 0.1 g, a test portion of about 6 g of the material, dissolve in water and dilute to 100 ml. Slowly pipette out 0.5 ml of this solution into 10 ml of the mercury (II) chloride solution in one of the Nessler cylinders. To 10 ml of the mercury (II) chloride solution contained in the second Nessler cylinder, slowly add 0.25 ml of the standard thiosulphate solution. Allow both the cylinders to stand for 10 min without agitation, then carefully agitate to distribute the opalescence. Immediately examine, in the Nessler cylinders, the opalescence produced in the test and control solutions.

NOTE — If the solutions are allowed to stand for more than 15 min, secondary reactions may occur which will affect the result.

A-5.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the opalescence produced in the test with the material is not greater than that produced in the control test.

A-6 TEST FOR CLARITY OF 20 PERCENT AQUEOUS SOLUTION

A-6.1 Procedure

Dissolve 20 g of the material in a little quantity of water and make up the volume to 100 ml. The solution shall not have more than a pale yellow colour and shall be free from extraneous impurities other than slight flocculence.

A-7 DETERMINATION OF pH

A-7.1 Apparatus

A-7.1.1 Electronic pH Meter

Equipped with a glass electrode and standard reference electrode.

A-7.2 Procedure

Weigh, to the nearest 0.1 g, about 5 g of the sample, dissolve in about 80 ml of previously boiled water having a *pH* not less than 6.5, and make up to 100 ml. Measure the *pH* of this solution using the *pH* meter.

A-8 REACTION TO AMMONIACAL SILVER NITRATE SOLUTION**A-8.1 Apparatus**

A-8.1.1 *Nessler Cylinder* — 50-ml capacity.

A-8.2 Reagents

A-8.2.1 *Ammoniacal Silver Nitrate Solution*

Mix equal volumes of ammonia solution (approximately

3 N) and 0.1 N silver nitrate solution. When required, the solution shall be prepared freshly.

A-8.3 Procedure

Weigh to the nearest 0.1 g, about 2 g of the material and dissolve in 40 ml of water. Divide into two equal portions. To one, add 10 ml of ammoniacal silver nitrate solution and mix well. To the other, the control solution, add 5 ml of ammonia solution and 5 ml of water and mix well. Allow each to stand for 2 min. Compare the colours and turbidities of the two solutions.

A-8.3.1 The material shall be taken to have passed the test if the colour or the turbidity produced with the material is not greater than that produced in the control test.

ANNEX B

(*Clause 3.3*)

DETERMINATION OF PARTICLE SIZE**B-1 PROCEDURE**

Assemble the 850-micron and 150-micron IS Sieves in the order of aperture sizes together with bottom receiver. Place 10.0 g of the material on 850-micron IS Sieve. Shake the sieves for 15 min by means of a mechanical sieve vibrator. Alternatively, the assembled sieves may be shaken by hand with frequent tapping

at an angle of 30° to the horizontal on a felt pad, and with occasional rotation, in order to ensure even distribution of the material.

B-1.1 Weigh separately the material retained and passing through each sieve and express it as percentage of the material taken for the test.

ANNEX C

(*Clause 5.1*)

SAMPLING OF POTASSIUM METABISULPHITE, PHOTOGRAPHIC GRADE**C-1 GENERAL REQUIREMENTS OF SAMPLING**

C-1.0 In drawing, preparing, storing and handling test samples, the precautions given in C-1.1 to C-1.7 shall be observed.

C-1.1 Samples shall not be taken at a place exposed to weather.

C-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

C-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

C-1.4 The samples shall be placed in suitable, clean, dry and air-tight opaque glass or plastics container.

C-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

C-1.6 Each sample containers shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture.

C-1.7 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

C-2 SCALE OF SAMPLING

C-2.1 Lot

All the containers in a single consignment of the material and drawn from a single batch of manufacture shall constitute a lot.

C-2.1.1 Samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification.

C-2.2 The number (*n*) of containers to be selected from a lot depends on the size of the lot (*N*) and shall be in accordance with col 1 and 2 of Table 2.

C-2.3 The containers to be selected for sampling shall be drawn at random from the lot. For random sampling procedures, guidance may be taken from IS 4905.

C-3 PREPARATION OF TEST SAMPLES

C-3.1 Draw with an appropriate sampling instrument 50 g of potassium metabisulphite from different parts

Table 2 Scale of Sampling of Containers
(Clause C-2.2)

Lot Size	Number of Containers to be Selected in the Sample
(<i>N</i>)	(<i>n</i>)
(1)	(2)
Up to 50	3
51 to 100	4
101 to 300	5
301 and above	7

of each container selected. This portion shall be transferred to a suitable sample container. From each of the sample containers approximately equal quantities of the material shall be taken and mixed together to form a composite sample weighing about 100 g. The remaining material in each of the sample container shall constitute the individual sample.

C-4 CRITERIA FOR CONFORMITY

C-4.1 Test for potassium metabisulphite and iron content shall be conducted on individual samples. The lot shall be considered as conforming to these requirements if all the individual samples pass the test.

C-4.2 Tests for all other characteristics shall be carried out on composite sample. The lot shall be declared as conforming to the specification if the test results on the composite sample satisfy the corresponding requirements.

ANNEX D
(Foreword)
COMMITTEE COMPOSITION

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

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